10563207

Connecting via Winsock to STN

Welcome to STN International! Enter x:x

LOGINID:ssspta1612bxr

PASSWORD:
TERMINAL (ENTER 1, 2, 3, OR ?):2

* * * * * * * * * * Welcome to STN International * * * * * * * * * *

NEWS 1		Web Page for STN Seminar Schedule - N. America
NEWS 2	NOV 21	CAS patent coverage to include exemplified prophetic substances identified in English-, French-, German-, and Japanese-language basic patents from 2004-present
NEWS 3	NOV 26	MARPAT enhanced with FSORT command
NEWS 4	NOV 26	CHEMSAFE now available on STN Easy
NEWS 5	NOV 26	Two new SET commands increase convenience of STN searching
NEWS 6	DEC 01	ChemPort single article sales feature unavailable
NEWS 7	DEC 12	GBFULL now offers single source for full-text coverage of complete UK patent families
NEWS 8	DEC 17	Fifty-one pharmaceutical ingredients added to PS
NEWS 9	JAN 06	The retention policy for unread STNmail messages will change in 2009 for STN-Columbus and STN-Tokyo
NEWS 10	JAN 07	WPIDS, WPINDEX, and WPIX enhanced Japanese Patent Classification Data
NEWS 11	FEB 02	Simultaneous left and right truncation (SLART) added for CERAB, COMPUAB, ELCOM, and SOLIDSTATE
NEWS 12	FEB 02	GENBANK enhanced with SET PLURALS and SET SPELLING
NEWS 13	FEB 06	Patent sequence location (PSL) data added to USGENE
NEWS 14	FEB 10	COMPENDEX reloaded and enhanced
NEWS 15	FEB 11	WTEXTILES reloaded and enhanced
NEWS 16	FEB 19	New patent-examiner citations in 300,000 CA/CAplus patent records provide insights into related prior art
NEWS 17	FEB 19	Increase the precision of your patent queries -- use terms from the IPC Thesaurus, Version 2009.01
NEWS 18	FEB 23	Several formats for image display and print options discontinued in USPATFULL and USPAT2
NEWS 19	FEB 23	MEDLINE now offers more precise author group fields and 2009 MeSH terms
NEWS 20	FEB 23	TOXCENTER updates mirror those of MEDLINE - more precise author group fields and 2009 MeSH terms
NEWS 21	FEB 23	Three million new patent records blast AEROSPACE into STN patent clusters
NEWS 22	FEB 25	USGENE enhanced with patent family and legal status display data from INPADOCDB
NEWS 23	MAR 06	INPADOCDB and INPAFAMDB enhanced with new display formats
NEWS 24	MAR 11	EPFULL backfile enhanced with additional full-text

Updated Search

```
                  applications and grants
 NEWS 25  MAR 11  ESBIOBASE reloaded and enhanced

 NEWS EXPRESS JUNE 27 08 CURRENT WINDOWS VERSION IS V8.3,
              AND CURRENT DISCOVER FILE IS DATED 23 JUNE 2008.

 NEWS HOURS    STN Operating Hours Plus Help Desk Availability
 NEWS LOGIN    Welcome Banner and News Items
 NEWS IPC8     For general information regarding STN implementation of IPC 8

Enter NEWS followed by the item number or name to see news on that
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* * * * * * * * * * * * * * * * STN  Columbus  * * * * * * * * * * * * * * * *

FILE 'HOME' ENTERED AT 16:04:31 ON 12 MAR 2009

=> file casreact
COST IN U.S. DOLLARS                                SINCE FILE      TOTAL
                                                         ENTRY    SESSION
FULL ESTIMATED COST                                       0.44       0.44

FILE 'CASREACT' ENTERED AT 16:05:42 ON 12 MAR 2009
USE IS SUBJECT TO THE TERMS OF YOUR CUSTOMER AGREEMENT
COPYRIGHT (C) 2009 AMERICAN CHEMICAL SOCIETY (ACS)

Copyright of the articles to which records in this database refer is
held by the publishers listed in the PUBLISHER (PB) field (available
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26, 1996), unless otherwise indicated in the original publications.

FILE CONTENT:1840 - 8 Mar 2009 VOL 150 ISS 11

New CAS Information Use Policies, enter HELP USAGETERMS for details.

************************************************************
*                                                          *
*     CASREACT now has more than 16.5 million reactions    *
*                                                          *
************************************************************

CASREACT contains reactions from CAS and from: ZIC/VINITI database
(1974-1999) provided by InfoChem; INPI data prior to 1986;
Biotransformations database compiled under the direction of
Professor Dr. Klaus Kieslich; organic reactions, portions copyright
1996-2006 John Wiley & Sons, Ltd., John Wiley and Sons, Inc.,
Organic Reactions Inc., and Organic Syntheses Inc. Reproduced under
license. All Rights Reserved.

This file contains CAS Registry Numbers for easy and accurate substance
```

identification.

=>
Uploading C:\Documents and Settings\brobinson1\My Documents\sdfndafjk.str

L1 STRUCTURE UPLOADED

=> s l1
SAMPLE SEARCH INITIATED 16:12:52 FILE 'CASREACT'
SCREENING COMPLETE - 0 REACTIONS TO VERIFY FROM 0 DOCUMENTS

100.0% DONE 0 VERIFIED 0 HIT RXNS 0 DOCS
SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE **COMPLETE**
BATCH **COMPLETE**
PROJECTED VERIFICATIONS: 0 TO 0
PROJECTED ANSWERS: 0 TO 0

L2 0 SEA SSS SAM L1 (0 REACTIONS)

=> s l1 full
THE ESTIMATED SEARCH COST FOR FILE 'CASREACT' IS 122.65 U.S. DOLLARS
DO YOU WANT TO CONTINUE WITH THIS REQUEST? (Y)/N or END:y
FULL SEARCH INITIATED 16:12:56 FILE 'CASREACT'
SCREENING COMPLETE - 0 REACTIONS TO VERIFY FROM 0 DOCUMENTS

100.0% DONE 0 VERIFIED 0 HIT RXNS 0 DOCS
SEARCH TIME: 00.00.01

L3 0 SEA SSS FUL L1 (0 REACTIONS)

=>
Uploading C:\Documents and Settings\brobinson1\My Documents\arae.str

L4 STRUCTURE UPLOADED

=> s l4
SAMPLE SEARCH INITIATED 16:14:27 FILE 'CASREACT'
SCREENING COMPLETE - 0 REACTIONS TO VERIFY FROM 0 DOCUMENTS

100.0% DONE 0 VERIFIED 0 HIT RXNS 0 DOCS
SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE **COMPLETE**
BATCH **COMPLETE**
PROJECTED VERIFICATIONS: 0 TO 0
PROJECTED ANSWERS: 0 TO 0

L5 0 SEA SSS SAM L4 (0 REACTIONS)

=> s l4 full
THE ESTIMATED SEARCH COST FOR FILE 'CASREACT' IS 122.65 U.S. DOLLARS
DO YOU WANT TO CONTINUE WITH THIS REQUEST? (Y)/N or END:y
FULL SEARCH INITIATED 16:14:31 FILE 'CASREACT'
SCREENING COMPLETE - 0 REACTIONS TO VERIFY FROM 0 DOCUMENTS

10563207

100.0% DONE 0 VERIFIED 0 HIT RXNS 0 DOCS
SEARCH TIME: 00.00.01

L6 0 SEA SSS FUL L4 (0 REACTIONS)

=>
Uploading C:\Documents and Settings\brobinson1\My Documents\araerty.str

L7 STRUCTURE UPLOADED

=> s l7
SAMPLE SEARCH INITIATED 16:15:53 FILE 'CASREACT'
SCREENING COMPLETE - 47 REACTIONS TO VERIFY FROM 6 DOCUMENTS

100.0% DONE 47 VERIFIED 5 HIT RXNS 2 DOCS
SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE **COMPLETE**
BATCH **COMPLETE**
PROJECTED VERIFICATIONS: 529 TO 1351
PROJECTED ANSWERS: 2 TO 124

L8 2 SEA SSS SAM L7 (5 REACTIONS)

=> file reg
COST IN U.S. DOLLARS SINCE FILE ENTRY TOTAL SESSION
FULL ESTIMATED COST 253.46 253.90

FILE 'REGISTRY' ENTERED AT 16:16:04 ON 12 MAR 2009
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STRUCTURE FILE UPDATES: 11 MAR 2009 HIGHEST RN 1119363-64-2
DICTIONARY FILE UPDATES: 11 MAR 2009 HIGHEST RN 1119363-64-2

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REGISTRY includes numerically searchable data for experimental and
predicted properties as well as tags indicating availability of
experimental property data in the original document. For information
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http://www.cas.org/support/stngen/stndoc/properties.html

=>

Updated Search

10563207

Uploading C:\Documents and Settings\brobinson1\My Documents\araerty.str

L9 STRUCTURE UPLOADED

=> file hcaplus
COST IN U.S. DOLLARS SINCE FILE ENTRY 0.48 TOTAL SESSION 254.38
FULL ESTIMATED COST

FILE 'HCAPLUS' ENTERED AT 16:16:19 ON 12 MAR 2009
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FILE COVERS 1907 - 12 Mar 2009 VOL 150 ISS 11
FILE LAST UPDATED: 11 Mar 2009 (20090311/ED)

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substance identification.

=> s l9/prep
SEARCH PROFILE NOT SUPPORTED FOR AUTOMATED SEARCH AND CROSSOVER
The search profile contains L-numbers or saved item names that include
chemical substance terms, chemical structures, or structure screen
sets. If you are in a single file environment using the CA file (CA,
HCA, ZCA, CAPLUS, HCAPLUS, ZCAPLUS), enter HELP FIRST at an arrow
prompt (=>) for information about the REG1stRY automated search and
crossover feature. REG1stRY supports the following search profiles:

Example 1:
=> ACT SCRSTR/Q
L3 STR
L4 SCR 2127
L5 QUE L3 NOT L4

These searches are supported:
S L5/REG
S SCRSTR/Q/REG

Updated Search

```
   S  (L3 NOT L4)/REG

   These searches are not supported:
   S  L5
   S  SCRSTR/Q

Example 2:
   => ACT SCRSTR2/Q
   L6              STR
   L7              SCR 2127
   L8              QUE  L6
   L9              QUE  L7
   L10             QUE  L8 NOT L9

   This search is supported:
   S  (L6 NOT L7)/REG

   These searches are not supported:
   S  L10
   S  L10/REG
   S  SCRSTR2/Q
   S  SCRSTR2/Q/REG
   S  L8 NOT L9
   S  (L8 NOT L9)/REG

=> file reg
COST IN U.S. DOLLARS                            SINCE FILE      TOTAL
                                                     ENTRY    SESSION
FULL ESTIMATED COST                                   2.85     257.23

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DICTIONARY FILE UPDATES:  11 MAR 2009  HIGHEST RN 1119363-64-2

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predicted properties as well as tags indicating availability of
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on property searching in REGISTRY, refer to:

http://www.cas.org/support/stngen/stndoc/properties.html

=>
```

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Uploading C:\Documents and Settings\brobinson1\My Documents\araerty.str

L10 STRUCTURE UPLOADED

=> s l10
SAMPLE SEARCH INITIATED 16:16:49 FILE 'REGISTRY'
SAMPLE SCREEN SEARCH COMPLETED - 40 TO ITERATE

100.0% PROCESSED 40 ITERATIONS 12 ANSWERS
SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE **COMPLETE**
BATCH **COMPLETE**
PROJECTED ITERATIONS: 421 TO 1179
PROJECTED ANSWERS: 33 TO 447

L11 12 SEA SSS SAM L10

=> s l10 full
THE ESTIMATED SEARCH COST FOR FILE 'REGISTRY' IS 185.40 U.S. DOLLARS
DO YOU WANT TO CONTINUE WITH THIS REQUEST? (Y)/N or END:y
FULL SEARCH INITIATED 16:16:53 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - 693 TO ITERATE

100.0% PROCESSED 693 ITERATIONS 201 ANSWERS
SEARCH TIME: 00.00.01

L12 201 SEA SSS FUL L10

=> file hcaplus
COST IN U.S. DOLLARS SINCE FILE ENTRY TOTAL SESSION
FULL ESTIMATED COST 185.88 443.11

FILE 'HCAPLUS' ENTERED AT 16:16:56 ON 12 MAR 2009
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FILE COVERS 1907 - 12 Mar 2009 VOL 150 ISS 11
FILE LAST UPDATED: 11 Mar 2009 (20090311/ED)

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10563207

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This file contains CAS Registry Numbers for easy and accurate
substance identification.

=> s l12
L13   2418 L12

=> s l12/prep
          2418 L12
       4736176 PREP/RL
L14   371 L12/PREP
                (L12 (L) PREP/RL)

=> file reg
COST IN U.S. DOLLARS   SINCE FILE   TOTAL
                                                     ENTRY   SESSION
FULL ESTIMATED COST   2.85   445.96

FILE 'REGISTRY' ENTERED AT 16:17:06 ON 12 MAR 2009
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on property searching in REGISTRY, refer to:

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=>
Uploading C:\Documents and Settings\brobinson1\My Documents\3207.str

L15   STRUCTURE UPLOADED

=> s l15
SAMPLE SEARCH INITIATED 16:18:41 FILE 'REGISTRY'
SAMPLE SCREEN SEARCH COMPLETED -   28 TO ITERATE

100.0% PROCESSED   28 ITERATIONS   0 ANSWERS

Updated Search

10563207

SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE **COMPLETE**
BATCH **COMPLETE**
PROJECTED ITERATIONS: 243 TO 877
PROJECTED ANSWERS: 0 TO 0

L16 0 SEA SSS SAM L15

=> s l15 full
THE ESTIMATED SEARCH COST FOR FILE 'REGISTRY' IS 185.40 U.S. DOLLARS
DO YOU WANT TO CONTINUE WITH THIS REQUEST? (Y)/N or END:y
FULL SEARCH INITIATED 16:18:45 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - 449 TO ITERATE

100.0% PROCESSED 449 ITERATIONS 8 ANSWERS
SEARCH TIME: 00.00.01

L17 8 SEA SSS FUL L15

=> file hcaplus
COST IN U.S. DOLLARS SINCE FILE ENTRY TOTAL SESSION
FULL ESTIMATED COST 186.84 632.80

FILE 'HCAPLUS' ENTERED AT 16:19:01 ON 12 MAR 2009
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FILE COVERS 1907 - 12 Mar 2009 VOL 150 ISS 11
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This file contains CAS Registry Numbers for easy and accurate
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=> s l17
L18 13 L17

Updated Search

10563207

```
=> s l17/prep
            13 L17
       4736176 PREP/RL
L19         12 L17/PREP
                (L17 (L) PREP/RL)

=> file reg
COST IN U.S. DOLLARS                                SINCE FILE      TOTAL
                                                         ENTRY    SESSION
FULL ESTIMATED COST                                       2.85     635.65

FILE 'REGISTRY' ENTERED AT 16:19:11 ON 12 MAR 2009
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DICTIONARY FILE UPDATES:  11 MAR 2009  HIGHEST RN 1119363-64-2

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experimental property data in the original document.  For information
on property searching in REGISTRY, refer to:

http://www.cas.org/support/stngen/stndoc/properties.html

=>
Uploading C:\Documents and Settings\brobinson1\My Documents\awr.str

L20     STRUCTURE UPLOADED

=> s l20 full
THE ESTIMATED SEARCH COST FOR FILE 'REGISTRY' IS 185.40 U.S. DOLLARS
DO YOU WANT TO CONTINUE WITH THIS REQUEST? (Y)/N or END:y
FULL SEARCH INITIATED 16:20:32 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED -      693 TO ITERATE

100.0% PROCESSED      693 ITERATIONS                          201 ANSWERS
SEARCH TIME: 00.00.01

L21         201 SEA SSS FUL L20

=> file hcaplus
COST IN U.S. DOLLARS                                SINCE FILE      TOTAL
                                                         ENTRY    SESSION
```

Updated Search

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FULL ESTIMATED COST 186.36 822.01

FILE 'HCAPLUS' ENTERED AT 16:20:35 ON 12 MAR 2009
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FILE LAST UPDATED: 11 Mar 2009 (20090311/ED)

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This file contains CAS Registry Numbers for easy and accurate
substance identification.

```
=> s l21/prep
          2418 L21
       4736176 PREP/RL
L22         371 L21/PREP
                  (L21 (L) PREP/RL)

=> d his

     (FILE 'HOME' ENTERED AT 16:04:31 ON 12 MAR 2009)

     FILE 'CASREACT' ENTERED AT 16:05:42 ON 12 MAR 2009
L1                STRUCTURE UPLOADED
L2              0 S L1
L3              0 S L1 FULL
L4                STRUCTURE UPLOADED
L5              0 S L4
L6              0 S L4 FULL
L7                STRUCTURE UPLOADED
L8              2 S L7

     FILE 'REGISTRY' ENTERED AT 16:16:04 ON 12 MAR 2009
L9                STRUCTURE UPLOADED

     FILE 'HCAPLUS' ENTERED AT 16:16:19 ON 12 MAR 2009
```

Updated Search

10563207

```
     FILE 'REGISTRY' ENTERED AT 16:16:27 ON 12 MAR 2009
L10             STRUCTURE UPLOADED
L11          12 S L10
L12         201 S L10 FULL

     FILE 'HCAPLUS' ENTERED AT 16:16:56 ON 12 MAR 2009
L13        2418 S L12
L14         371 S L12/PREP

     FILE 'REGISTRY' ENTERED AT 16:17:06 ON 12 MAR 2009
L15             STRUCTURE UPLOADED
L16           0 S L15
L17           8 S L15 FULL

     FILE 'HCAPLUS' ENTERED AT 16:19:01 ON 12 MAR 2009
L18          13 S L17
L19          12 S L17/PREP

     FILE 'REGISTRY' ENTERED AT 16:19:11 ON 12 MAR 2009
L20             STRUCTURE UPLOADED
L21         201 S L20 FULL

     FILE 'HCAPLUS' ENTERED AT 16:20:35 ON 12 MAR 2009
L22         371 S L21/PREP

=> s l17/rct
            13 L17
       3232764 RCT/RL
L23           3 L17/RCT
                  (L17 (L) RCT/RL)

=> d his

     (FILE 'HOME' ENTERED AT 16:04:31 ON 12 MAR 2009)

     FILE 'CASREACT' ENTERED AT 16:05:42 ON 12 MAR 2009
L1              STRUCTURE UPLOADED
L2            0 S L1
L3            0 S L1 FULL
L4              STRUCTURE UPLOADED
L5            0 S L4
L6            0 S L4 FULL
L7              STRUCTURE UPLOADED
L8            2 S L7

     FILE 'REGISTRY' ENTERED AT 16:16:04 ON 12 MAR 2009
L9              STRUCTURE UPLOADED

     FILE 'HCAPLUS' ENTERED AT 16:16:19 ON 12 MAR 2009

     FILE 'REGISTRY' ENTERED AT 16:16:27 ON 12 MAR 2009
L10             STRUCTURE UPLOADED
L11          12 S L10
L12         201 S L10 FULL

     FILE 'HCAPLUS' ENTERED AT 16:16:56 ON 12 MAR 2009
```

Updated Search

```
L13        2418 S L12
L14         371 S L12/PREP

     FILE 'REGISTRY' ENTERED AT 16:17:06 ON 12 MAR 2009
L15             STRUCTURE UPLOADED
L16           0 S L15
L17           8 S L15 FULL

     FILE 'HCAPLUS' ENTERED AT 16:19:01 ON 12 MAR 2009
L18          13 S L17
L19          12 S L17/PREP

     FILE 'REGISTRY' ENTERED AT 16:19:11 ON 12 MAR 2009
L20             STRUCTURE UPLOADED
L21         201 S L20 FULL

     FILE 'HCAPLUS' ENTERED AT 16:20:35 ON 12 MAR 2009
L22         371 S L21/PREP
L23           3 S L17/RCT

=> s l23 and l22
L24           0 L23 AND L22

=> file reg
COST IN U.S. DOLLARS                            SINCE FILE      TOTAL
                                                     ENTRY    SESSION
FULL ESTIMATED COST                                   2.85     824.86

FILE 'REGISTRY' ENTERED AT 16:21:06 ON 12 MAR 2009
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STRUCTURE FILE UPDATES:   11 MAR 2009  HIGHEST RN 1119363-64-2
DICTIONARY FILE UPDATES:  11 MAR 2009  HIGHEST RN 1119363-64-2

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REGISTRY includes numerically searchable data for experimental and
predicted properties as well as tags indicating availability of
experimental property data in the original document.  For information
on property searching in REGISTRY, refer to:

http://www.cas.org/support/stngen/stndoc/properties.html

=> d l15
L15 HAS NO ANSWERS
L15             STR
```
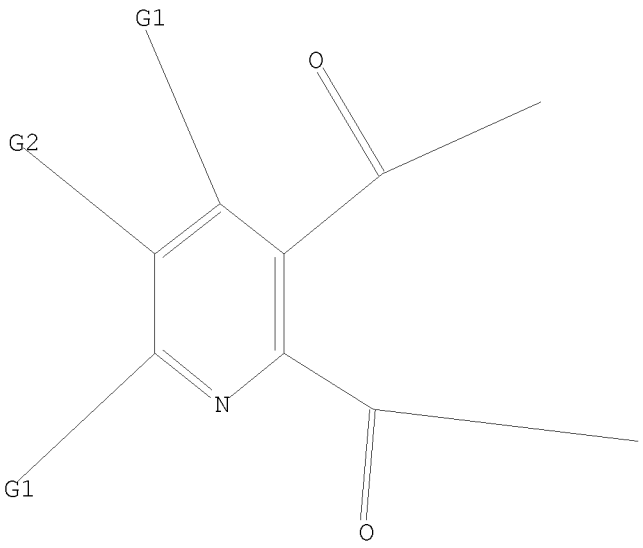

10563207



G1 H,Ak,Ph

G2 X,Ak,Ph,H

Structure attributes must be viewed using STN Express query preparation.

=> d his

```
     (FILE 'HOME' ENTERED AT 16:04:31 ON 12 MAR 2009)

     FILE 'CASREACT' ENTERED AT 16:05:42 ON 12 MAR 2009
L1                STRUCTURE UPLOADED
L2              0 S L1
L3              0 S L1 FULL
L4                STRUCTURE UPLOADED
L5              0 S L4
L6              0 S L4 FULL
L7                STRUCTURE UPLOADED
L8              2 S L7

     FILE 'REGISTRY' ENTERED AT 16:16:04 ON 12 MAR 2009
L9                STRUCTURE UPLOADED

     FILE 'HCAPLUS' ENTERED AT 16:16:19 ON 12 MAR 2009

     FILE 'REGISTRY' ENTERED AT 16:16:27 ON 12 MAR 2009
L10               STRUCTURE UPLOADED
L11            12 S L10
L12           201 S L10 FULL

     FILE 'HCAPLUS' ENTERED AT 16:16:56 ON 12 MAR 2009
L13          2418 S L12
L14           371 S L12/PREP
```

Updated Search

10563207

```
     FILE 'REGISTRY' ENTERED AT 16:17:06 ON 12 MAR 2009
L15             STRUCTURE UPLOADED
L16           0 S L15
L17           8 S L15 FULL

     FILE 'HCAPLUS' ENTERED AT 16:19:01 ON 12 MAR 2009
L18          13 S L17
L19          12 S L17/PREP

     FILE 'REGISTRY' ENTERED AT 16:19:11 ON 12 MAR 2009
L20             STRUCTURE UPLOADED
L21         201 S L20 FULL

     FILE 'HCAPLUS' ENTERED AT 16:20:35 ON 12 MAR 2009
L22         371 S L21/PREP
L23           3 S L17/RCT
L24           0 S L23 AND L22

     FILE 'REGISTRY' ENTERED AT 16:21:06 ON 12 MAR 2009

=> file hcaplus
COST IN U.S. DOLLARS                            SINCE FILE      TOTAL
                                                     ENTRY    SESSION
FULL ESTIMATED COST                                   0.48     825.34

FILE 'HCAPLUS' ENTERED AT 16:21:17 ON 12 MAR 2009
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FILE COVERS 1907 - 12 Mar 2009  VOL 150 ISS 11
FILE LAST UPDATED: 11 Mar 2009  (20090311/ED)

HCAplus now includes complete International Patent Classification (IPC)
reclassification data for the third quarter of 2008.

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http://www.cas.org/legal/infopolicy.html

This file contains CAS Registry Numbers for easy and accurate
substance identification.

=> s l22 and saponif?
         19164 SAPONIF?
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Updated Search

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         55927 SAPON
            92 SAPONS
         55973 SAPON
                 (SAPON OR SAPONS)
         29762 SAPOND
             1 SAPONDS
         29763 SAPOND
                 (SAPOND OR SAPONDS)
          3227 SAPONG
         90651 SAPONIF?
                 (SAPONIF? OR SAPON OR SAPOND OR SAPONG)
L25         15 L22 AND SAPONIF?

=> s l25 and oxidiz?
        448349 OXIDIZ?
L26          1 L25 AND OXIDIZ?

=> d l26, ibib abs, 1

L26  ANSWER 1 OF 1  HCAPLUS  COPYRIGHT 2009 ACS on STN
ACCESSION NUMBER:        2005:55206  HCAPLUS
DOCUMENT NUMBER:         142:155823
TITLE:                   In-situ treatment of pyridine-2,3-dicarboxylic acid
                         esters with an oxidizing agent for the
                         removal of impurities
INVENTOR(S):             Levy, Michael A.
PATENT ASSIGNEE(S):      BASF Aktiengesellschaft, Germany
SOURCE:                  PCT Int. Appl., 23 pp.
                         CODEN: PIXXD2
DOCUMENT TYPE:           Patent
LANGUAGE:                English
FAMILY ACC. NUM. COUNT:  1
PATENT INFORMATION:

     PATENT NO.           KIND   DATE        APPLICATION NO.        DATE
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     WO 2005005391         A1    20050120    WO 2004-EP6893         20040625
         W:  AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH,
             CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD,
             GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC,
             LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI,
             NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY,
             TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW
         RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM,
             AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK,
             EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE,
             SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE,
             SN, TD, TG
     CA 2530110            A1    20050120    CA 2004-2530110        20040625
     EP 1644333            A1    20060412    EP 2004-740304         20040625
         R:  AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,
             IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, PL, SK, HR
     CN 1816526            A     20060809    CN 2004-80018858       20040625
     BR 2004012091         A     20060905    BR 2004-12091          20040625
     IN 2006CN00420        A     20070518    IN 2006-CN420          20060201
     US 20070185331        A1    20070809    US 2006-563207         20060630
```

10563207

PRIORITY APPLN. INFO.: US 2003-484485P P 20030702
WO 2004-EP6893 W 20040625
OTHER SOURCE(S): MARPAT 142:155823
AB A method for the in-situ treatment of a pyridine-2,3-dicarboxylic acid ester with an oxidizing agent, such as hydrogen peroxide, to improve product quality is described. The method for the in-situ removal of impurities from a sapond. solution of pyridine-2,3-dicarboxylic acid esters comprises providing a solution of a pyridine-2,3-dicarboxylic acid ester, sapong. the solution with a base to form the corresponding pyridine-2,3-dicarboxylic acid salt, reacting the solution with an oxidizing agent in an amount effective to remove impurities, acidifying the solution with an acid to convert the pyridine-2,3-dicarboxylic acid into the corresponding pyridine-2,3-dicarboxylic acid, and collecting a purified solution comprising a pyridine-2,3-dicarboxylic acid (e.g., 5-methyl-2,3-pyridinedicarboxylic acid).
REFERENCE COUNT: 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

=> d his

(FILE 'HOME' ENTERED AT 16:04:31 ON 12 MAR 2009)

FILE 'CASREACT' ENTERED AT 16:05:42 ON 12 MAR 2009
L1 STRUCTURE UPLOADED
L2 0 S L1
L3 0 S L1 FULL
L4 STRUCTURE UPLOADED
L5 0 S L4
L6 0 S L4 FULL
L7 STRUCTURE UPLOADED
L8 2 S L7

FILE 'REGISTRY' ENTERED AT 16:16:04 ON 12 MAR 2009
L9 STRUCTURE UPLOADED

FILE 'HCAPLUS' ENTERED AT 16:16:19 ON 12 MAR 2009

FILE 'REGISTRY' ENTERED AT 16:16:27 ON 12 MAR 2009
L10 STRUCTURE UPLOADED
L11 12 S L10
L12 201 S L10 FULL

FILE 'HCAPLUS' ENTERED AT 16:16:56 ON 12 MAR 2009
L13 2418 S L12
L14 371 S L12/PREP

FILE 'REGISTRY' ENTERED AT 16:17:06 ON 12 MAR 2009
L15 STRUCTURE UPLOADED
L16 0 S L15
L17 8 S L15 FULL

FILE 'HCAPLUS' ENTERED AT 16:19:01 ON 12 MAR 2009
L18 13 S L17
L19 12 S L17/PREP

Updated Search

10563207

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L20             STRUCTURE UPLOADED
L21         201 S L20 FULL

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L22         371 S L21/PREP
L23           3 S L17/RCT
L24           0 S L23 AND L22

     FILE 'REGISTRY' ENTERED AT 16:21:06 ON 12 MAR 2009

     FILE 'HCAPLUS' ENTERED AT 16:21:17 ON 12 MAR 2009
L25          15 S L22 AND SAPONIF?
L26           1 S L25 AND OXIDIZ?

=> s l25 not l26
L27         14 L25 NOT L26

=> s l27 and levy, m?/au
          2253 LEVY, M?/AU
L28          0 L27 AND LEVY, M?/AU

=> d l27, ibib abs hitstr, 1-14

L27  ANSWER 1 OF 14  HCAPLUS  COPYRIGHT 2009 ACS on STN
ACCESSION NUMBER:        2006:669115  HCAPLUS
DOCUMENT NUMBER:         145:230277
TITLE:                   Water-Mediated Multicenter Synthon and Aromatic C-H
                         → N Isostructurality
AUTHOR(S):               Babu, N. Jagadeesh; Nangia, Ashwini
CORPORATE SOURCE:        School of Chemistry, University of Hyderabad,
                         Hyderabad, 500 046, India
SOURCE:                  Crystal Growth & Design (2006), 6(8), 1753-1756
                         CODEN: CGDEFU; ISSN: 1528-7483
PUBLISHER:               American Chemical Society
DOCUMENT TYPE:           Journal
LANGUAGE:                English
AB   A strong, cooperative O-H•••O hydrogen bond network directs
     isostructurality in pyrazinetetracarboxylic acid, pyridinetetracarboxylic
     acid, and pyromellitic acid dihydrates.  H2O and COOH groups reorganize
     their H bonding groups in an invariant network leading to
     Ow-H•••N ↔ C-H•••Ow mimicry.  The degree of
     similarity is related to the size of the multicenter synthon and the
     strength of the Oacid-H•••Owater hydrogen bond.
IT   905564-94-5P, Pyridine-2,3,5,6-tetracarboxylic acid dihydrate
     RL: PRP (Properties); SPN (Synthetic preparation); PREP
     (Preparation)
        (crystallog.; water-mediated multicenter synthon and aromatic C-H →
        N isostructurality)
RN   905564-94-5  HCAPLUS
CN   2,3,5,6-Pyridinetetracarboxylic acid, hydrate (1:2)  (CA INDEX NAME)
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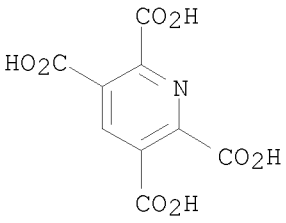
10563207



●2 H_2O

REFERENCE COUNT: 32 THERE ARE 32 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L27 ANSWER 2 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN
ACCESSION NUMBER: 1997:436082 HCAPLUS
DOCUMENT NUMBER: 127:50632
ORIGINAL REFERENCE NO.: 127:9661a,9664a
TITLE: Preparation of cyclic amic acid derivatives as inhibitors of protein-farnesyl transferase and antitumor agents
INVENTOR(S): Iwasawa, Yoshikazu; Aoyama, Tetsuya; Kawakami, Kumiko; Arai, Sachie; Satoh, Toshihiko; Monden, Yoshiaki
PATENT ASSIGNEE(S): Banyu Pharmaceuticals Co., Ltd., Japan
SOURCE: PCT Int. Appl., 100 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9717321	A1	19970515	WO 1996-JP3239	19961106
W: AU, CA, CN, JP, KR, US				
RW: AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
AU 9675051	A	19970529	AU 1996-75051	19961106
PRIORITY APPLN. INFO.:			JP 1995-313625 A	19951107
			WO 1996-JP3239 W	19961106

OTHER SOURCE(S): MARPAT 127:50632
GI

* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

AB Compds. of general formula [I; wherein Ar1, Ar2 and Ar3 = aryl or heteroaryl; Cy = aryl, heteroaryl, alicyclic; Q = (CH2)m (m = an integer of 1 to 6) or (CH2)n-W-(CH2)p (W = oxygen, sulfur, vinylene or ethynylene; n, p = an integer of 0 to 3); R1 = H, halo, OH, (un)substituted lower alkyl or alkoxy; R2, R7, R8 = H, halo, OH, lower alkyl or alkoxy; R3, R4 = H, halo, OH, NH2, NO2, cyano, CO2H, lower alkoxycarbonyl, CONH2, lower

Updated Search

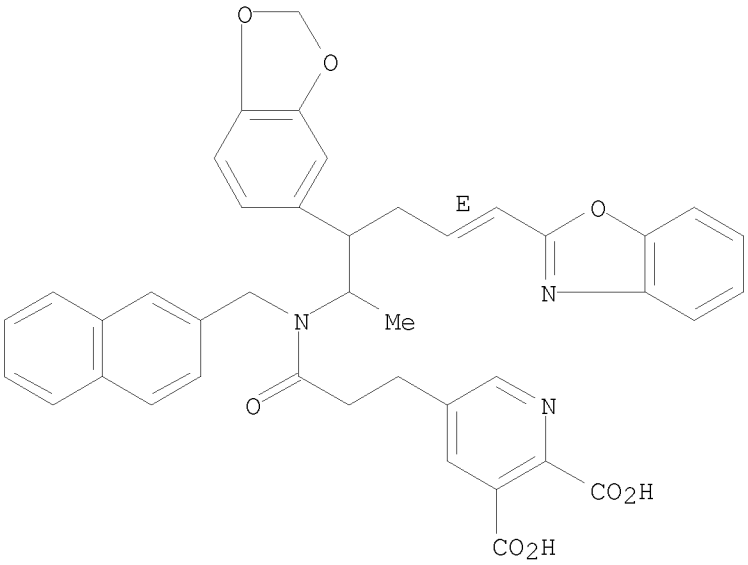
alkylcarbamoyl, lower alkyl, hydroxyalkyl, fluoroalkyl, or alkoxy; R5 = lower alkyl; R6 = H, lower alkyl; R9, R10 = H, OH, lower alkyl; R11 = OH, CO2H, lower alkyl, hydroxyalkyl, or alkoxy; p, n = an integer of 0 to 2; m = 0 or 1] or pharmaceutically acceptable salts and esters thereof are prepared An antitumor agent containing I as the active ingredient is claimed. Thus, a 5-carbamoyl-1,3-dioxolane-2,2,4-tricarboxylic acid derivative (II; R = CHO, R12 = Me, R13 = Et) (preparation given) underwent Wittig reaction with 2-benzoxazolylmethyltriphenylphosphonium chloride using NaH in THF followed by sapon. with LiOH in aqueous THF and acidification with 1 N aqueous HCl to give II (R = Q, R12 = R13 = H). The latter compound in vitro showed IC50 of 0.1 nM for inhibiting protein-farnesyl transferase and 3.6 nM for inhibiting the farnesylation of Ras protein in activated ras gene-transformed NIH3T3 cells and in vivo inhibited the proliferation of activated human Ha-ras-transformed cells (NIH/ras) transplanted in mice by 23, 41, and 82% at 20, 40, and 80 mg/kg i.p.

IT 191088-25-2P
RL: BAC (Biological activity or effector, except adverse); BSU (Biological study, unclassified); SPN (Synthetic preparation); THU (Therapeutic use); BIOL (Biological study); PREP (Preparation); USES (Uses)
(preparation of cyclic amic acid derivs. as inhibitors of protein-farnesyl transferase and antitumor agents)

RN 191088-25-2 HCAPLUS

CN 2,3-Pyridinedicarboxylic acid, 5-[3-[[(4E)-2-(1,3-benzodioxol-5-yl)-5-(2-benzoxazolyl)-1-methyl-4-penten-1-yl](2-naphthalenylmethyl)amino]-3-oxopropyl]- (CA INDEX NAME)

Double bond geometry as shown.



REFERENCE COUNT: 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L27 ANSWER 3 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN
ACCESSION NUMBER: 1994:270130 HCAPLUS
DOCUMENT NUMBER: 120:270130

Updated Search

10563207

ORIGINAL REFERENCE NO.: 120:47851a,47854a
TITLE: 5,6-disubstituted-3-pyridylmethylammonium halide compounds useful for the preparation of 5-(substituted methyl)-2,3-pyridinedicarboxylic acids
INVENTOR(S): Strong, Henry L.
PATENT ASSIGNEE(S): American Cyanamid Co., USA
SOURCE: U.S., 10 pp. Cont.-in-part of U.S. Ser. No. 812,520, abandoned.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 5288866	A	19940222	US 1992-960749	19921014
AT 151752	T	19970515	AT 1992-119537	19921116
ES 2100261	T3	19970616	ES 1992-119537	19921116
SK 280466	B6	20000214	SK 1992-3665	19921215
SK 280477	B6	20000214	SK 1998-1400	19921215
CZ 286513	B6	20000517	CZ 1992-3665	19921215
JP 05255257	A	19931005	JP 1992-353923	19921216
JP 3107672	B2	20001113		
IL 104134	A	19970610	IL 1992-104134	19921217
CA 2085802	A1	19930621	CA 1992-2085802	19921218
CA 2085802	C	20030916		
BR 9205097	A	19930622	BR 1992-5097	19921218
AU 9230280	A	19930624	AU 1992-30280	19921218
AU 652874	B2	19940908		
ZA 9209877	A	19930702	ZA 1992-9877	19921218
HU 64052	A2	19931129	HU 1992-4021	19921218
HU 217563	B	20000228		
HU 218004	B	20000528	HU 1996-2838	19921218
CN 1094398	A	19941102	CN 1993-105332	19930430
CN 1042333	C	19990303		
RU 2090558	C1	19970920	RU 1993-5302	19930511
US 5378843	A	19950103	US 1993-156205	19931122
US 5545835	A	19960813	US 1994-334297	19941104
CZ 286519	B6	20000517	CZ 1997-1082	19970409
CN 1190094	A	19980812	CN 1998-103644	19980113
CN 1067379	C	20010620		

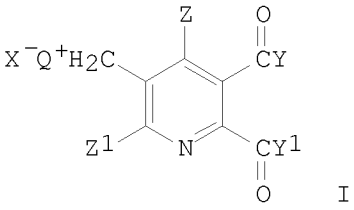
PRIORITY APPLN. INFO.:

US 1991-812520	B2	19911220
US 1992-960749	A	19921014
CS 1992-3665	A	19921215
HU 1992-4021	A	19921218
US 1993-156205	A3	19931122

OTHER SOURCE(S): CASREACT 120:270130; MARPAT 120:270130
GI

Updated Search

10563207



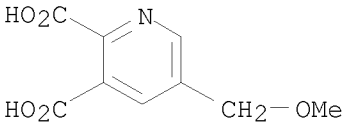
I

AB A method for the preparation of 5,6-disubstituted-3-pyridylmethylammonium halide compds. I (Z = H, halo; Z1 = H, halo, cyano, nitro; X = Cl, Br, iodo, alkylsulfonyl; Y and Y1 = alkoxy, amino; Q = cyclic or hydrocarbyl ammonium) is provided. I can be used for the preparation of 5-(substituted methyl)-2,3-pyridinedicarboxylic acids. Thus, bromination of di-Me 5-methyl-2,3-pyridinedicarboxylate with NBS in the presence of 2,2'-azobisisobutyronitrile in CCl4 gave 57% di-Me 5-(bromomethyl)-2,3-pyridinedicarboxylate which on treatment with amines in EtOH gave I.

IT 143382-03-0P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

RN 143382-03-0 HCAPLUS

CN 2,3-Pyridinedicarboxylic acid, 5-(methoxymethyl)- (CA INDEX NAME)



REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L27 ANSWER 4 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1991:228750 HCAPLUS

DOCUMENT NUMBER: 114:228750

ORIGINAL REFERENCE NO.: 114:38581a,38584a

TITLE: Process for preparing pyridine-2,3-dicarboxylic acids and esters as agrochemical and pharmaceutical intermediates

INVENTOR(S): Yamashita, Takaharu; Kodama, Mitsuhiro; Shimada, Shouzo

PATENT ASSIGNEE(S): Sugai Chemical Industry Co., Ltd., Japan

SOURCE: U.S., 10 pp. Cont.-in-part of U.S. Ser. No. 139,641, abandoned.
CODEN: USXXAM

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 2

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE

Updated Search

US 4973695	A	19901127	US 1989-308524		19890210
JP 63301867	A	19881208	JP 1988-836		19880106
JP 2561500	B2	19961211			
PRIORITY APPLN. INFO.:			JP 1987-915	A	19870106
			JP 1987-290389	A	19871116
			US 1987-139641	B2	19871230

OTHER SOURCE(S): CASREACT 114:228750; MARPAT 114:228750

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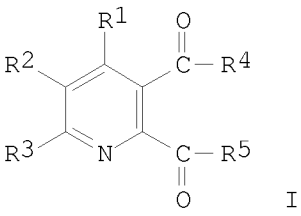
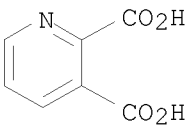


I

AB The title compds. [I; R1, R3 = H, alkyl; R2 = H, alkyl, (un)substituted phenylalkyl; R4, R5 = alkoxy], useful as pharmaceutical and agrochem. intermediates, e.g., for herbicidal 2-(2-imidazolin-2-yl)pyridine-3-carboxylic acids (no data), were prepared by a process comprising cyclocondensation of propenals and analogs R1CH:CR2COR3 with α-halooxalacetate esters R4COCHXCOCOR5 (X = halo) and NH3, possibly in the presence of a secondary or tertiary amine or an ammonium salt, in an aprotic H2O-immiscible solvent at 20-200°, at atmospheric or elevated pressure. Thus, 44.5 g di-Et α-chlorooxalacetate in 250 mL PhCl was added dropwise into a mixture of 21.0 g 2-ethyl-2-propenal in 350 mL PhCl at 88-94° over 40 min while NH3(g) was bubbled through the system. After the addition was completed the temperature was raised to 115° and NH3(g) bubbled for 4 addnl. h to give 76.5% title compound I [R1 = R3 = H, R2 = Et (II; R4 = R5 = EtO)] which (10.3 g) was sapond. by refluxing for 3.5 h with 48% aqueous NaOH in PhMe/H2O and acidified to give 5.9 g title acid II (R4 = R5 = OH).

IT 89-00-9P, 2,3-Pyridinedicarboxylic acid 53636-65-0P
53636-70-7P 102268-15-5P,
5-Ethylpyridine-2,3-dicarboxylic acid 133787-56-1P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of, as herbicide intermediate)

RN 89-00-9 HCAPLUS
CN 2,3-Pyridinedicarboxylic acid (CA INDEX NAME)



RN 53636-65-0 HCAPLUS
CN 2,3-Pyridinedicarboxylic acid, 5-methyl- (CA INDEX NAME)

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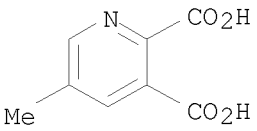
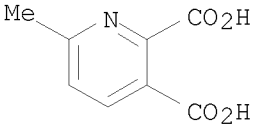
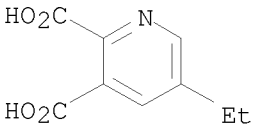


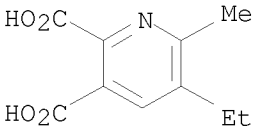
RN 53636-70-7 HCAPLUS
CN 2,3-Pyridinedicarboxylic acid, 6-methyl- (CA INDEX NAME)



RN 102268-15-5 HCAPLUS
CN 2,3-Pyridinedicarboxylic acid, 5-ethyl- (CA INDEX NAME)



RN 133787-56-1 HCAPLUS
CN 2,3-Pyridinedicarboxylic acid, 5-ethyl-6-methyl- (CA INDEX NAME)



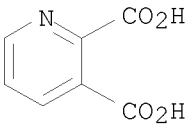
REFERENCE COUNT: 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L27 ANSWER 5 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN
ACCESSION NUMBER: 1988:221572 HCAPLUS
DOCUMENT NUMBER: 108:221572
ORIGINAL REFERENCE NO.: 108:36367a,36370a
TITLE: 2-Aza-2,4-cyclopentadienone. Existence and reactivity
AUTHOR(S): Gavina, F.; Costero, A. M.; Andreu, M. R.; Carda, M.; Luis, S. V.
CORPORATE SOURCE: Dep. Quim. Org., Univ. Valencia, Valencia, Spain
SOURCE: Journal of the American Chemical Society (1988), 110(12), 4017-18
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: English

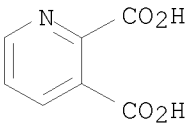
Updated Search

10563207

OTHER SOURCE(S): CASREACT 108:221572
AB The elusive species 2-aza-2,4-cyclopentadienone (I) is generated from an insol. polymeric precursor. The liberated intermediate acts either as a diene or a dienophile in Diels-Alder reactions. Thus, treatment of the polymeric 5-sulfonate of 3-pyrrolidin-2-one with the polymeric monoester of HO2CC.tplbond.CCO2H at 100° in DMSO gives a polymeric adduct, which, after sapon., gives 2,3-pyridinedicarboxylic acid. This acid results from the reaction of I as a diene with the C.tplbond.C bond, followed by CO extrusion and aromatization. I reacts as a dienophile with the polymeric ester of 2-furoic acid to give, after basic hydrolysis and Clemmensen reduction, δ-coniceine.
IT 89-00-9P, 2,3-Pyridinedicarboxylic acid
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and esterification of, with chloromethylated polymer)
RN 89-00-9 HCAPLUS
CN 2,3-Pyridinedicarboxylic acid (CA INDEX NAME)



IT 89-00-9DP, 2,3-Pyridinedicarboxylic acid, polymer-bound monoester
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and sapon. of)
RN 89-00-9 HCAPLUS
CN 2,3-Pyridinedicarboxylic acid (CA INDEX NAME)



L27 ANSWER 6 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN
ACCESSION NUMBER: 1988:204620 HCAPLUS
DOCUMENT NUMBER: 108:204620
ORIGINAL REFERENCE NO.: 108:33629a,33632a
TITLE: Preparation and testing of arylimidazoles as herbicides
INVENTOR(S): Astles, David Phillip; Flood, Andrew
PATENT ASSIGNEE(S): Shell Internationale Research Maatschappij B. V., Neth.
SOURCE: Brit. UK Pat. Appl., 17 pp.
CODEN: BAXXDU
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1

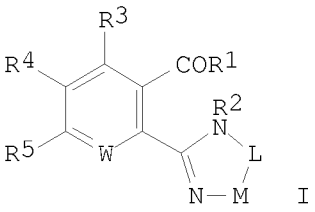
Updated Search

10563207

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 2192877	A	19880127	GB 1986-17898	19860722

PRIORITY APPLN. INFO.: GB 1986-17898 19860722
OTHER SOURCE(S): CASREACT 108:204620; MARPAT 108:204620
GI

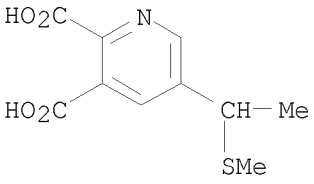


I

AB The title compds. (I; R1 = OR8, alkyl, alkenyl, alkynyl, cycloalkyl, Ph, furyl, PhCH2; R2 = H, acyl; R1R2 = bond; R3, R5 = H, halo, NO2, cyano, Q; R4 = H, halo, OH, NO2, Q; R6 = alkyl, cycloalkyl; R7 = alkyl, cycloalkyl, alkenyl, Ph, PhCH2; R8 = H, salt-forming cation; W = N, CH; one of L, M = CO, the other = CR6R7; Q= XYZC; X = cyano, thiol, amino, oximino, etc.; Y = H, alkyl, X; Z = H, alkyl) were prepared as herbicides. Di-Me 5-ethylpyridine-2,3-dicarboxylate was successively photobrominated with NBS, condensed with NaSMe, sapond. with aqueous NaOH, refluxed with Ac2O to yield an anhydride, and condensed with 2-amino-2,3-dimethylbutyramide to give 2-[(1-carbonyl-1,2-dimethylpropyl)carbonyl]-[5-[1-methylthio)ethyl]nicotinic acid, which was cyclized in 3 M NaOH to give 2-(5-isopropyl-5-methyl-4-oxo-2-imidazolin-2-yl)-5-[1-(methylthio)-ethyl]nicotinic acid (II). At 1 kg/ha preemergent, II gave complete control of Echinochloa crusgalli.

IT 114311-42-1P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and conversion of, to anhydride)

RN 114311-42-1 HCAPLUS
CN 2,3-Pyridinedicarboxylic acid, 5-[1-(methylthio)ethyl]- (CA INDEX NAME)



L27 ANSWER 7 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN
ACCESSION NUMBER: 1988:94556 HCAPLUS
DOCUMENT NUMBER: 108:94556
ORIGINAL REFERENCE NO.: 108:15555a,15558a
TITLE: Preparation of 2-(imidazol-2-yl)pyridine-3-carboxylic

Updated Search

10563207

acid derivatives as herbicides

INVENTOR(S): Numata, Tatsuo; Hatanaka, Masataka; Watanabe, Junichi; Igai, Takashi; Nawamaki, Tsutomu; Hattori, Kenji

PATENT ASSIGNEE(S): Nissan Chemical Industries, Ltd., Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 18 pp.
CODEN: JKXXAF

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

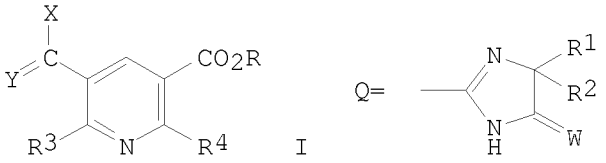
FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 62174069	A	19870730	JP 1986-13040	19860124
JP 07000611	B	19950111		

PRIORITY APPLN. INFO.: JP 1986-13040 19860124

GI



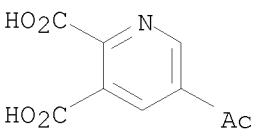
I

AB The title compds. [I; R4 = Q; W = O, S; X = H, (halo)alkyl, alkylsulfonylmethyl, alkoxymethyl, alkylthiomethyl, PhCH2, (un)substituted Ph or pyridyl; Y = O, S, monosubstituted NH, disubstituted CH2; R = H, (dialkyl)NH, (un)substituted alkyl, (un)substituted alkenyl, alkynyl, (un)substituted cycloalkyl, (un)substituted NH4+, alkali or alkaline earth metal; R1 = alkyl; R2 = (cyclo)alkyl; CR1R2 = (alkyl)cycloalkylene; R3 = H, halo, alkyl(thio), alkoxy, phenoxy, (halo)alkoxy, alkylsulfonyl], useful as herbicides, were prepared A mixture of 2.0 g 5-ethenyl-6-methylpyridine-2,3-dicarboxylic acid anhydride and 1.5 g H2NCMe(CHMe2)CONH2 in pyridine was vigorously stirred overnight to give a crude I [R4 = CONHCMe(CHMe2)CONH2, R = X = H, R3 = Me, Y = CH2] which was treated with aqueous NaOH at 80° for 3 h to give, after acidification with aqueous HCl, 1.1 g I (R4 = Q, W = O, X = R = H, Y = CH2, R1 = R3 = Me, R2 = CHMe2) (II). Postemergence treatment with II at 0.63 kg/ha completely controlled all 12 weeds tested, e.g., Echinochloa crus-galli showing no damage to soybean.

IT 113051-96-0P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and acid anhydride formation of)

RN 113051-96-0 HCAPLUS

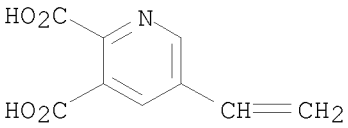
CN 2,3-Pyridinedicarboxylic acid, 5-acetyl- (CA INDEX NAME)



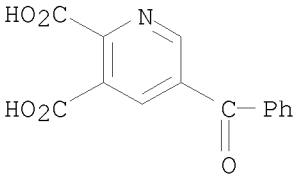
Updated Search

10563207

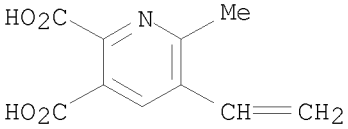
IT 113052-03-2P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and amidation of, with aminobutanamide derivative, imidazolylpyridine derivative from)
RN 113052-03-2 HCAPLUS
CN 2,3-Pyridinedicarboxylic acid, 5-ethenyl- (CA INDEX NAME)



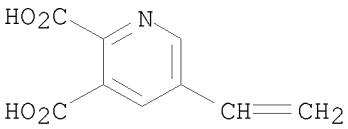
IT 113051-97-1P 113052-02-1P 113052-03-2P
113052-04-3P 113052-05-4P 113052-06-5P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of, as intermediate for herbicidal imidazolylpyridine derivative)
RN 113051-97-1 HCAPLUS
CN 2,3-Pyridinedicarboxylic acid, 5-benzoyl- (CA INDEX NAME)



RN 113052-02-1 HCAPLUS
CN 2,3-Pyridinedicarboxylic acid, 5-ethenyl-6-methyl- (CA INDEX NAME)



RN 113052-03-2 HCAPLUS
CN 2,3-Pyridinedicarboxylic acid, 5-ethenyl- (CA INDEX NAME)

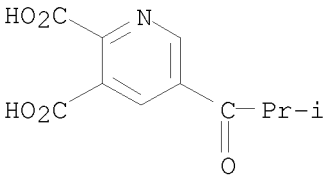


RN 113052-04-3 HCAPLUS

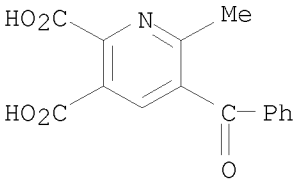
Updated Search

10563207

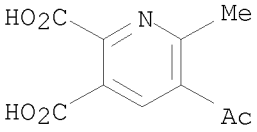
CN 2,3-Pyridinedicarboxylic acid, 5-(2-methyl-1-oxopropyl)- (CA INDEX NAME)



RN 113052-05-4 HCAPLUS
CN 2,3-Pyridinedicarboxylic acid, 5-benzoyl-6-methyl- (CA INDEX NAME)



RN 113052-06-5 HCAPLUS
CN 2,3-Pyridinedicarboxylic acid, 5-acetyl-6-methyl- (CA INDEX NAME)



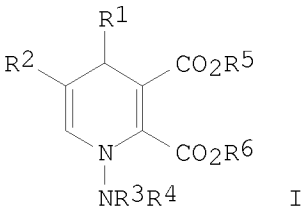
L27 ANSWER 8 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN
ACCESSION NUMBER: 1988:37663 HCAPLUS
DOCUMENT NUMBER: 108:37663
ORIGINAL REFERENCE NO.: 108:6299a,6302a
TITLE: New substituted dihalopyridines and procedure for their preparation as well as their further conversion into pyridinedicarboxylic acid diesters
INVENTOR(S): Astles, David Phillip; Flood, Andrew
PATENT ASSIGNEE(S): Shell Internationale Research Maatschappij B. V., Neth.
SOURCE: Ger. Offen., 5 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3707530	A1	19870917	DE 1987-3707530	19870309

Updated Search

NL 8700324	A	19871001	NL 1987-324	19870211
FR 2595354	A1	19870911	FR 1987-3163	19870309
FR 2595354	B1	19931126		
JP 62212368	A	19870918	JP 1987-52246	19870309
JP 08032683	B	19960329		
GB 2188318	A	19870930	GB 1987-5472	19870309
GB 2188318	B	19900214		
CH 671762	A5	19890929	CH 1987-871	19870309
BE 1003158	A5	19911217	BE 1987-229	19870310
JP 08198852	A	19960806	JP 1995-258314	19950912
JP 2631644	B2	19970716		

PRIORITY APPLN. INFO.: GB 1986-5868 A 19860310
OTHER SOURCE(S): CASREACT 108:37663
GI

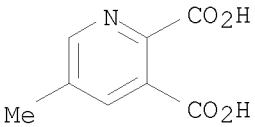


I

AB Dihydropyridines I [R1 = H, (un)substituted allyl or cycloalkyl; R2 (un)substituted alkyl or cycloalkyl; R3, R4 independently = R2; R5, R6 independently = (un)substituted alkyl, cycloalkyl, alkenyl, alkynyl, aryl, or aralkyl] useful as intermediates for herbicidal pyridylimidazolinones, were prepared by reaction of R1CH:CR2CH:NNR3R4 with R5O2C.tplbond.CO2R6. MeO2CC.tplbond.CCO2Me and H2C:CMeCH:NNMe2 were refluxed in PhMe 1 h to give I (R1 = H, R2-R6 = Me), dehydrogenation of which gave di-Me 5-methyl-2,3-pyridinedicarboxylate. Sapon. gave the corresponding dicarboxylic acid, dehydration of which, with Ac2O gave 5-methyl-2,3-pyridinedicarboxylic anhydride.

IT 53636-65-0P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of, as herbicide intermediate)

RN 53636-65-0 HCAPLUS
CN 2,3-Pyridinedicarboxylic acid, 5-methyl- (CA INDEX NAME)



REFERENCE COUNT: 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

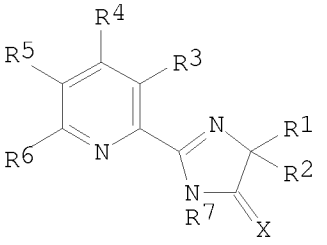
L27 ANSWER 9 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN
ACCESSION NUMBER: 1987:213943 HCAPLUS
DOCUMENT NUMBER: 106:213943

10563207

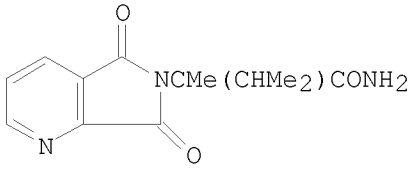
ORIGINAL REFERENCE NO.: 106:34721a,34724a
TITLE: Herbicidal 2-(2-imidazolin-2-yl)pyridine derivatives
INVENTOR(S): Los, Marinus
PATENT ASSIGNEE(S): American Cyanamid Co., USA
SOURCE: Brit. UK Pat. Appl., 361 pp.
CODEN: BAXXDU
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 2174395	A	19861105	GB 1986-11303	19860509

PRIORITY APPLN. INFO.: GB 1986-11303 19860509
OTHER SOURCE(S): CASREACT 106:213943; MARPAT 106:213943
GI



I

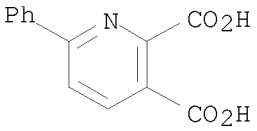


II

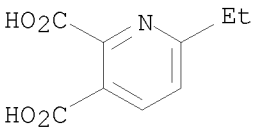
AB The title compds. [I; R1 = C1-4 alkyl; R2 = C1-4 alkyl, C3-6 cycloalkyl; R1R2 = (Me-substituted) C2-5 alkylene; R3 = (un)modified CO2H, acyl, HOCH2, carboxyalkyl, oxazolidinyl, (substituted) alkenyl, alkynyl, cycloalkyl, etc; R4 = H, halo, OH, Me; R5, R6 = H, halo, (substituted) C1-6 alkyl, hydroxyalkyl, C1-6 alkoxy, C1-4 alkylthio, PhO, NO2, cyano, amino; R5R6 = atoms to complete a fused, (un)subst. aromatic ring; R7 = H, (substituted) acyl, sulfonyl; X = O, S] and related compds. were prepared as herbicides. Thus, pyrrolopyridineacetamide II was treated successively with diazabicycloundcene and MeOH to give I (R1 = Me, R2 = Me2CH, R3 = CO2Me, R4-R7 = H, X = O). This was sapond. and treated with Et3N to give I.Et3N (R1 = Me, R2 = Me2CH, R3 = CO2H, R4-R7 = H, X = O) (III). At 0.032 kg/ha III gave a complete kill of quackgrass.

IT 39633-01-7P 90376-91-3P 90376-92-4P
90376-93-5P 90376-94-6P 90376-96-8P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and dehydration of, quinolinic anhydride derivative by)
RN 39633-01-7 HCAPLUS
CN 2,3-Pyridinedicarboxylic acid, 6-phenyl- (CA INDEX NAME)

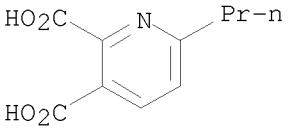
Updated Search



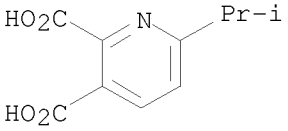
RN 90376-91-3 HCAPLUS
CN 2,3-Pyridinedicarboxylic acid, 6-ethyl- (CA INDEX NAME)



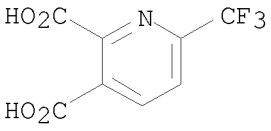
RN 90376-92-4 HCAPLUS
CN 2,3-Pyridinedicarboxylic acid, 6-propyl- (CA INDEX NAME)



RN 90376-93-5 HCAPLUS
CN 2,3-Pyridinedicarboxylic acid, 6-(1-methylethyl)- (CA INDEX NAME)

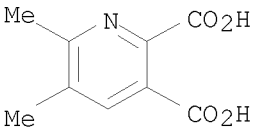


RN 90376-94-6 HCAPLUS
CN 2,3-Pyridinedicarboxylic acid, 6-(trifluoromethyl)- (CA INDEX NAME)

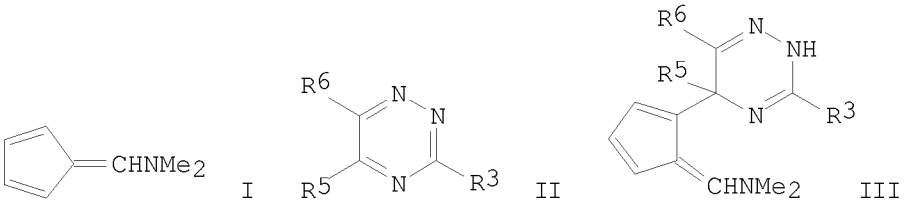


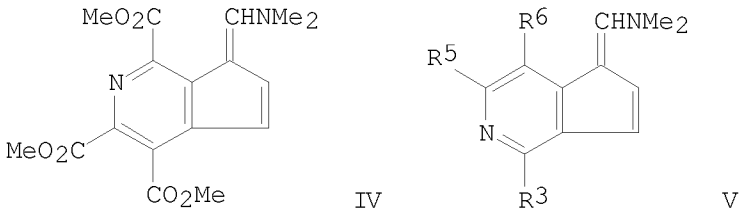
RN 90376-96-8 HCAPLUS
CN 2,3-Pyridinedicarboxylic acid, 5,6-dimethyl- (CA INDEX NAME)

10563207



L27 ANSWER 10 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN
ACCESSION NUMBER: 1985:523452 HCAPLUS
DOCUMENT NUMBER: 103:123452
ORIGINAL REFERENCE NO.: 103:19749a,19752a
TITLE: Chemistry of 1,2,4-triazines, XII. Cycloaddition reactions of azabenzenes, XVII. Reactions of 1,2,4-triazines with 6-(dimethylamino)pentafulvene
AUTHOR(S): Neunhoeffer, Hans; Bachmann, Michael
CORPORATE SOURCE: Inst. Org. Chem. Biochem., Tech. Hochsch. Darmstadt, Darmstadt, D-6100, Fed. Rep. Ger.
SOURCE: Liebigs Annalen der Chemie (1985), (6), 1263-6
CODEN: LACHDL; ISSN: 0170-2041
DOCUMENT TYPE: Journal
LANGUAGE: German
OTHER SOURCE(S): CASREACT 103:123452
GI

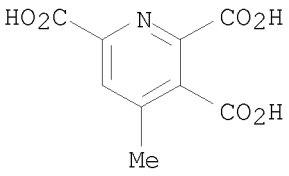




AB Pentafulvene I reacted with triazines II (R3, R5, R6 = Me, CO2Me, CO2Me; Me, CO2Et, CO2Et; CO2Me, CO2Me, CO2Me; CO2Me, Ph, H; CO2Me, Ph, Ph) either via addition to C5 of II to give pentafulvenyltriazines III or by a [4+2]cycloaddn. to give pyrindenes IV/V. No [6+4] cycloaddn. between I and II was observed There was no reaction between I and II (R3, R5, R6 = Ph, H, H; H, Ph, H; Ph, Ph, Ph; Me, Me, Me) in boiling dioxane or boiling xylene; in diglycine, only tar-like decomposition products were obtained.
IT 98166-53-1P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP

Updated Search

(Preparation); RACT (Reactant or reagent)
(preparation and decarboxylation of)
RN 98166-53-1 HCAPLUS
CN 2,3,6-Pyridinetricarboxylic acid, 4-methyl- (CA INDEX NAME)



L27 ANSWER 11 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN
ACCESSION NUMBER: 1956:69416 HCAPLUS
DOCUMENT NUMBER: 50:69416
ORIGINAL REFERENCE NO.: 50:13015b-i,13016a-d
TITLE: Synthesis in the 4-azafluorene group. I. Synthesis of 4-azafluorene and its 3-methyl derivative
AUTHOR(S): Chatterjea, J. N.; Prasad, K.
CORPORATE SOURCE: Sci. Coll., Patna
SOURCE: Journal of the Indian Chemical Society (1955), 32, 371-82
CODEN: JICSAH; ISSN: 0019-4522
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 50:69416
AB A solution of 4 g. 2-hydroxymethyleneindan-1-one, 2.1 g. cyanoacetamide, and 0.5 g. piperidine (I) in aqueous EtOH was kept at 50-5° 48 h., the mixture filtered, to the filtrate concentrated in vacuo was added 4-5 mL. more I, the mixture allowed to stand at 50° 24 days, and the dark brown solid filtered off, giving 3-oxo-2-cyano-4a-hydroxy-2,3,4,4a-tetrahydro-4-azafluorene (II), yellow crystals, m. 318-20° (decomposition) (from AcOH). II (3.6 g.) heated in a sealed tube at 170° 15 h. with 6 mL. fuming HCl and filtered gave 2.4 g. of 3-hydroxy-4-azafluorene-HCl (III), m. 302-6°, yellow prismatic needles from EtOH containing a few drops of HCl, which on being heated in NaOH solution gave on cooling colorless needles of the Na salt of III which were filtered off and treated with a large volume of H2O to yield III, yellow needles, m. 300-302°. A mixture of 1.4 g. III.HCl, 8 mL. POCl3, and 8 g. PCl5 was refuxed 96 h., evaporated in vacuo, the residue treated with ice, made alkaline with Na2CO3, extracted with Et2O, and the extract dried with K2CO3 and evaporated The alkaline solution on acidification gave 0.8 g. unchanged III, which was retreated with POCl3 and PCl5 giving a total of 0.35 g. crude 3-chloro-4-azafluorene (IV), chromatographed on Al2O3, eluted with petr. ether, giving 0.13 g. IV m. 92-3° (from petr. ether), colorless prisms, λ 254 and 311 mμ, ε 8700 and 18,000 resp.; IR spectrum given. Reduction of IV in NaOEt solution with Raney Ni gave a trace of 4-azafluorene (V). Reduction of IV with HI and red P, and the product made basic, steam distilled and extracted with Et2O gave a trace of V (picrate, m. about 200°). A solution of 4.8 g.

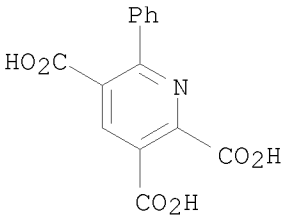
β-phenyl-β-aminoacrylonitrile and 8 g. Et ethoxymethyleneoxalacetate in 6 mL. AcOH heated 1.5 h. on a H2O-bath and evaporated in vacuo gave 5.9 g. di-Et 3-cyano-2-phenylpyridine-5,6-dicarboxylate (VI), silky needles, m. 92-3° (from EtOH). A mixture of 3.2 g. VI and 1.6 g. NaOH in 12 mL. 30 percent EtOH refluxed 20-24 h., filtered, and acidified with HCl, gave 2.4 g. 2-phenylpyridine-3,5,6-tricarboxylic acid (VII), needles from H2O, m. 256-7° with frothing at 153-60° (picrate, m. 274-6°). VI sapond. 4-5 h. gave 3-carbamoyl-2-phenylpyridine-5,6-dicarboxylic acid, prismatic needles from H2O, m. 295° with frothing at 160-6°. VI sapond. with 2 equivs. of alc. KOH gave 3-cyano-2-phenylpyridine-5,6-dicarboxylic acid, m. 202-4° (from H2O) with frothing at 152-6°. VII (1.5 g.) refluxed 1.5 h. with 10 mL. SOCl2, evaporated, the residue treated at 60° 4 h. with 25 mL. PhNO2 and 3.0 g. AlCl3, allowed to stand overnight, decomposed with ice and HCl, steam distilled, filtered, and the filtrate concentrated and filtered again gave 0.8 g. 4-azafluorenone-2,3-dicarboxylic acid (VIII), light yellow rectangular plates, m. 292° (from H2O). Decarboxylation of VIII by heating in 50-mg. batches gave 12 mg. 4-azafluorenone (IX), long flat needles, m. 139°, from H2O, identical with IX prepared by CrO3 oxidation of 4-azafluorene (X) IX picrate, m. 197° (from EtOH); IX semicarbazone, colorless needles, m. 257-8° (from EtOH); IX oxime, colorless prisms, m. 245-6° (from EtOH)]. Reduction of 0.1 g. IX with 5 mL. HI and 0.2 g. red P gave X.HI which on dissolving in H2O and basifying gave X, colorless prisms, m. 93° (from petr. ether) [picrate, m. 215°; picrolonate, yellow needles from BuOH, m. 243-4° (decomposition)]. UV spectra in EtOH, PO4 buffer pH 7, and 0.1N HCl, and IR spectrum are given. A mixture of 5.7 g. BzCH2CO2Et, 4.4 g. HC(OEt)3, and 6.1 g. Ac2O refluxed 1.5 h. and distilled gave 2.6 g. Et ethoxymethylene(benzoyl) acetate (XI), b4 190-2°. A mixture of 2.6 g. XI, 1.5 g. β-phenyl-β-aminoacrylonitrile, and 3 mL. Ac2O heated on a H2O bath 8 h. and evaporated gave Et 3-cyano-2,6-diphenylpyridine-5-carboxylate (XII), m. 145° (from EtOH). Sapon. of 0.2 g. XII 20 h. with 40% alc. NaOH, filtration, and acidification gave 0.15 g. 2,6-diphenylpyridine-3,5-dicarboxylic acid, yellow prisms from EtOH, m. 283°. A mixture of 8 g. β-phenyl-β-aminoacrylonitrile, 10.3 g. Et ethoxymethyleneacetoacetate, and 10 mL. AcOH was heated 3.5 h. on a steam bath, evaporated in vacuo, the residue warmed with 50 mL. EtOH, cooled, and filtered. From the solid, 3.3 g. Et 3-cyano-2-phenyl-6-methylpyridine-5-carboxylate (XIII), yellow rectangular plates from petr. ether, m. 86-8°, and 0.43 g. α-[(2-cyano-1-phenylvinylamino)methylene]acetoacetate (XIV), yellow rhombs from EtOH, m. 150-2°, were obtained. From the filtrate, 0.5 g. more XIII, and 3 compds. m. 240-1°, 296-8°, and 216° were obtained. Sapon. of XIII gave the corresponding acid (XV), yellow prisms from H2O, m. 211-12°, frothing at 139-40°, which was cyclized by treating the chloride with AlCl3, in PhNO2 to give VIII. Treatment of 2 g. VIII with 7 g. PCl5 in CHCl3, evaporation and treatment of a cold CS2 solution of the acid chloride with 6 g. AlCl3 gave 0.7 g. 3-methyl-4-azafluorenone-2-carboxylic acid (XVI), m. 248-9° (from H2O), which was decarboxylated by heating with Cu bronze to 3-methyl-4-azafluorenone, m. 120-1° (from petr. ether). Reduction of 0.4 g. XVI with 12 mL. HI and 0.5 g. red P gave 3-methyl-4-azafluorene-2-carboxylic acid-HI, converted to the free base (XVII), colorless needles from H2O, m. 260-2°. XVII was

decarboxylated by heating with Cu bronze to 3-methyl-4-azafluorene, m. 47-8° (picrate, yellow needles, m. 211°; picrolonate, yellow prisms from EtOH, m. 252-4°). XIV (0.35 g.) treated with 2 mL. cold H2SO2 gave 0.2 g. compound, m. 112-13° evolving gas above 170°, colorless flat needles from H2O, giving NH3 on being heated with alkali.

IT 856957-63-6P, 2,3,5-Pyridinetricarboxylic acid, 6-phenyl-
856957-64-7P, 2,3,5-Pyridinetricarboxylic acid, 6-phenyl-, picrate
860716-13-8P, Quinolinic acid, 5-carbamoyl-6-phenyl-
RL: PREP (Preparation)
(preparation of)

RN 856957-63-6 HCAPLUS

CN 2,3,5-Pyridinetricarboxylic acid, 6-phenyl- (CA INDEX NAME)

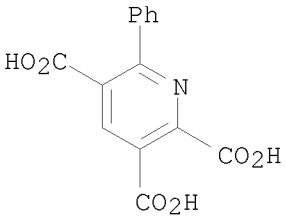


RN 856957-64-7 HCAPLUS

CN 2,3,5-Pyridinetricarboxylic acid, 6-phenyl-, picrate (5CI) (CA INDEX NAME)

CM 1

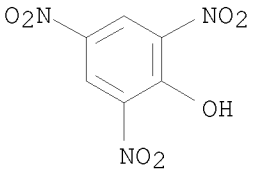
CRN 856957-63-6
CMF C14 H9 N O6



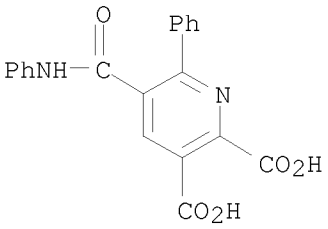
CM 2

CRN 88-89-1
CMF C6 H3 N3 O7

10563207



RN 860716-13-8 HCAPLUS
CN 2,3-Pyridinedicarboxylic acid, 6-phenyl-5-[(phenylamino)carbonyl]- (CA INDEX NAME)



L27 ANSWER 12 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN
ACCESSION NUMBER: 1955:64835 HCAPLUS
DOCUMENT NUMBER: 49:64835
ORIGINAL REFERENCE NO.: 49:12462f-i,12463a-i,12464a-f
TITLE: Utilization of n-alkyl methyl ketones in the Pfitzinger reaction
AUTHOR(S): Henze, Henry R.; Carroll, Donald W.
CORPORATE SOURCE: Univ. of Texas, Austin
SOURCE: Journal of the American Chemical Society (1954), 76, 4580-4
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 49:64835
AB A series of Pfitzinger condensations [cf. Pfitzinger, J. prakt. chemical [2], 56, 283(1897)] using n-alkyl Me ketones (alkyl = Me through C6H13) has been carried out. The unsym. ketones produced 2 isomeric cinchoninic acids (I), a 2-monosubstituted acid and a 2,3-disubstituted acid, the monosubstituted compound usually being formed in the larger amount A new sequence of syntheses has been developed in order to establish the structure of 1 series of these isomeric I. Isatin (II) (60 g.), 200 cc. 34% KOH in dilute EtOH, 88 g. EtAc, and 375 cc. H2O refluxed 72 hrs. with stirring, about 125 cc. liquid distilled off, the residue made faintly acidic and filtered, the filtrate acidified strongly, and the precipitate filtered off,
washed, and dried gave 70 g. crystalline mixture of isomeric I, decompose above 300° after sintering at 248°, which repeatedly recrystd.
from H2O and aqueous EtOH gave 2,3-dimethylcinchoninic acid (III), m. above 320° with rapid decomposition AcCO2H (25 g.) and 17 g. EtCHO in 100 cc. EtOH treated during 1.5 hrs. at about 5° with 27 g. PhNH2 in 50 cc. EtOH, the mixture warmed gently 3 hrs., refluxed 7 hrs., concentrated to 110°, and cooled, and the yellow solid deposit purified with EtOH

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and Me2CO gave 4.5 g. 2-ethylcinchoninic acid (IV), m. 180-1°. Samples of III and IV titrated with the aid of a Beckmann pH meter showed that the pH (8.2) at the neutralization point was identical for both acids. The composition of the crude mixture of III and IV obtained in the reaction was estimated to contain about 85% III, as determined by the m.p. behavior of a series of known mixts. of purified III and IV. II (60 g.), 120 g. PrAc, 200 cc. 34% KOH, and 370 cc. H2O gave in the usual manner 81 g. mixed acids, m. beginning at 131°, which recrystd. gave the major product, 2-propyl cinchoninic acid, silvery white plates, m. 159.0-9.5° (decomposition); the crude reaction product (30 g.) recrystd. repeatedly gave 7 g. 3-ethyl-2-methylcinchoninic acid (V), m. above 220°, which recrystd. from dioxane gave pure V, white friable powder, m. 257-8° (decomposition); the crude product contained 20-5% V. V heated gave with decarboxylation 3-ethyl-2-methylquinoline (VI), which yielded a picrate, m. 227-30° (decomposition). II (60 g.), 108 g. BuAc, 200 cc. 34% KOH, and 375 cc. H2O gave similarly 119 g. crude product, m. beginning at 121°, which recrystd. extensively gave 2-butylcinchoninic acid, white friable powder or very fine leaflets, m. 141-2°, which was decarboxylated to 2-butylquinoline, identified as the picrate, m. 162°. Fractional extraction of the crude reaction product gave a small amount of the isomeric 2-methyl-3-propylcinchoninic acid, snow-white powder, m. above 290° (decomposition). II (60 g.), 105 g. AmAc, 400 cc. 34% KOH, and 900 cc. H2O refluxed 78 hrs. with stirring gave similarly 79 g. crude mixed product, m. beginning about 125°, which recrystd. repeatedly gave 2-amylcinchoninic acid (VII), m. 135-6° (slight decomposition), previously regarded by Salzer, et al. (C.A. 43, 1415c), as 3-butyl-2-methylcinchoninic acid (VIII). VII decarboxylated, and the resulting product treated with picric acid gave 2-amylquinoline picrate, m. 103.0-3.5° (from aqueous EtOH). The crude product extracted with dioxane, and the residue from the extract recrystd. from EtOH gave 3-butyl-2-methylcinchoninic acid, granular white powder, m. 261-3° (decomposition), which constituted only about 5% of the crude product; a sample decarboxylated and treated with picric acid gave 3-butyl-2-methylquinoline picrate, fine yellow needles, m. 210-12° (decomposition). II (50 g.), 75 cc. C6H13Ac, 180 cc. 34% KOH, and 300 cc. H2O refluxed 96 hrs. with stirring gave 82 g. crude product, m. 136-40° (from H2O), which recrystd. repeatedly from MeOH gave 2-hexylcinchoninic acid (IX), m. 140-1°. IX decarboxylated and treated with picric acid in MeOH gave 2-hexylquinoline picrate, m. 110-12° (decomposition). 2-Methylcinchoninic acid (8 g.) in 200 cc. H2O containing 2 g. NaOH treated with stirring and heating on a steam cone with 67 g. KMnO4 in 1200 cc. H2O dropwise during 7 hrs., the mixture heated 70 hrs., treated with a few cc. EtOH to destroy the excess KMnO4, and filtered, the clear filtrate concentrated to about 500 cc., acidified with HNO3, and treated with 200 cc. aqueous hot solution containing 17 g. Cu(OAc)2, the pasty, blue precipitate filtered off, washed with about 500 cc. M AcOH, stirred while being treated with gaseous H2S, and filtered, the filtrate evaporated to dryness, the residue (about 2 g.) extracted with hot MeOH, the purplish gel which set to a solid (1.5 g.) powdered and extracted in a Soxhlet apparatus with EtOAc, and the extract evaporated gave 2,3,4,6-pyridinetetracarboxylic acid, light tan solid, m. 182-4°, expanded to resolidify and then melted with extensive decomposition at about 223-7°. (EtO)2CHCOCH2CO2Et (X) (45 g.) added to 4.8 g. Na in 100 cc. EtOH, the mixture treated during 70 min. at reflux temperature with 26 g.

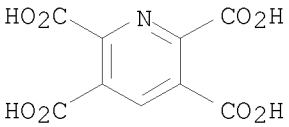
EtBr, refluxed 13 hrs., and filtered, the filtrate diluted with H2O and extracted with Et2O, and the extract dried with Na2SO4 and fractionated gave 38.5 g. (EtO)2CHCOCHEtCO2Et (XI), b5 118-21°, n20D 1.4270, d2020 1.0085, MRD 62.57, 63.61; it gave with aqueous FeCl3 a deep amber color within 1 min. XI (35.5 g.), 145 cc. 2N KOH, and 125 cc. MeOH refluxed 1 hr. with stirring, the MeOH distilled off, the alkaline solution extracted with Et2O, and the extract dried with Na2SO4 and fractionated gave 16.2 g. (EtO)2CHCOPr (XII), b9 78-9°, n20D 1.4130, d2020 0.9187, MRD 47.06; it gave with aqueous FeCl3 during 0.5 hr. a deep golden-brown color. XII treated with KCN and (NH4)2CO3 in aqueous EtOH gave 5-diethoxymethyl-5-propylhydantoin, m. 150°, XII gave a semicarbazone, m. 244° (decomposition); and a 2,4-dinitrophenylhydrazone, bright orange solid, m. 285-6° (decomposition). XII (10 g.) and 6.35 g. II in 22 cc. aqueous alc. KOH and 50 cc. H2O refluxed 72 hrs. with stirring, the mixture cooled, extracted with Et2O to recover a small amount of XII, acidified to precipitate inorg. salt and 3.2 g. II as an agglutinous red mass, and filtered, the filtrate basified with aqueous Na2CO3, concentrated to 75 cc. and acidified with concentrated HCl, and the resulting spongy, amorphous material crystallized from C6H6 gave 3.3 g. 2-diethoxymethyl-3-ethylcinchoninic acid (XIII), yellowish solid, m. 145-50° (recrystd. from C6H6 and Skellysolve A, fibrous white solid, m. 146.5°). XIII (750 mg.) heated about 4 hrs. with 60 cc. 0.25N H2SO4 on the steam cone, and the solution concentrated and chilled deposited about 500 mg. (88%) material, which recrystd. from hot dilute MeOH gave 3-ethyl-2-formylcinchoninic acid (XIV), white crystalline solid, m. 222-3° (decomposition); it gave a pos. Schiff test for aldehyde. XIV (0.5 g.), 3 g. amalgamated Zn, 12 cc. H2O, 3 cc. EtOH, and 15 cc. concentrated HCl refluxed 5.5 hrs., the liquid decanted, diluted with an equal volume H2O and sufficient aqueous NaOH to precipitate Zn(OH)2, and steam distilled to give 100 cc. distillate, the distillate extracted with Et2O, the extract dried and evaporated, and the small amount light brown oily residue treated with picric acid gave the picrate of VI, bright yellow crystals, m. 229.0-9.5° (decomposition); the mother liquor from the steam distillation gave 200 mg. brown material which could not be purified, since it charred on burning and underwent extensive decomposition at 250-4°; this product was possibly V. X alkylated in the usual manner with BuBr yielded (EtO)2CHCOHBuCO2Et (XV), b. 124-7°, n20D 1.4296, d2020 1.001, MRD 71.00; it gave a russet color with aqueous FeCl3 within 2 min. XV hydrolyzed with KOH in MeOH gave (EtO)2CHCOAm (XVI), b8-9 94°, b. 222°, n20D 1.427, d2020 0.912, MRD 56.90, which heated with KCN and (NH4)2CO3 in a sealed tube at 110° yielded 5-amyl-5-(dimethoxymethyl)hydantoin, white crystals, m. 119-20°. XVI did not give with II in alkaline solution a cinchoninic acid. Cl2CHCO2H was converted to (MeO)2CHCO2Et (XVII), b4-5 57-60°, n20D 1.4078, d2020 1.054, MRD 34.55. XVII condensed with EtOAc in the presence of Na yielded 76% (MeO)2CHCOCH2CO2Et (XVIII), b4 104.0 ± 0.5°, n20D 1.4286, d2020 1.084, MRD 45.15, which immediately gave a blood-red color when shaken with aqueous FeCl3. XVIII gave with H2NCONHNH2.HCl a compound which was apparently H2NCONHN:CHC(:NNHCONH2)CH2CO2Et, m. 227° with charring. XVIII

alkylated with NaOEt and BuBr gave (MeO)2CHCOCHBuCO2Et, b4-5 128.5-9.5°, n20D 1.4342, d2020 1.019, MRD 62.90; this sapond. with KOH in MeOH yielded 70% (MeO)2CHCOAm (XIX), b4-5 98-100°, n20D 1.4218, d2020 0.939, MRD 47.10. XIX gave a semicarbazone, white crystals, m. 241.2° (decomposition), and a 2,4-dinitrophenylhydrazone, fluffy bright orange powder, m. 185-6°. XIX gave with KCN and (NH4)2CO3 5-amyl-5-(dimethoxymethyl)hydantoin, m. 94-5°. XIX (9 g.), 5.4 g. II, 25 cc. 34% aqueous KOH, 45 cc. H2O, and 25 cc. EtOH refluxed 72 hrs. with stirring yielded 8 g. 3-butyl-2-dimethoxymethylcinchoninic acid (XX), m. 155-6° (from C6H6-Skellysolve A). XX (0.75 g.) in 75 cc. 0.4N H2SO4 heated 5 hrs. on the steam bath while adding from time to time small amts. H2O to keep the volume constant, the mixture cooled, and the resulting crude product (0.6 g., 94%) recrystd. from hot EtOH gave, 3-butyl-2-formylcinchoninic acid, small white crystals, m. 207° (decomposition), gave a pos. Schiff test and a raspberry-red with 2N aqueous KOH. XX in aqueous EtOH refluxed with concentrated HCl and amalgamated Zn, the resulting product dissolved in EtOH containing NaOH, the solution refluxed and neutralized, and the tan precipitate recrystd. from aqueous EtOH gave 3-butyl-2-methylcinchoninic acid, m. 261-4° with darkening. The substitution of the alkyl groups into the 2-, 3-, or 2,3-positions of cinchoninic acids did not significantly change the maximum or min. points of the ultraviolet absorption; the 2-formylcinchoninic acids exhibited a change, evidently due to a lengthening of the conjugation of the unsatn.

IT 14660-50-5P, 2,3,5,6-Pyridinetetracarboxylic acid
RL: PREP (Preparation)
(preparation of)

RN 14660-50-5 HCAPLUS

CN 2,3,5,6-Pyridinetetracarboxylic acid (CA INDEX NAME)



L27 ANSWER 13 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN

ACCESSION NUMBER: 1953:44619 HCAPLUS

DOCUMENT NUMBER: 47:44619

ORIGINAL REFERENCE NO.: 47:7512g-i

TITLE: Alkaloids of Makrotomia echoides. I. New alkaloid makrotomine and its structure

AUTHOR(S): Men'shikov, G. P.; Petrova, M. F.

CORPORATE SOURCE: S. Ordzhonikidze All-Union Chem.-Pharm. Research Inst.

SOURCE: Zhurnal Obshchei Khimii (1952), 22, 1457-61
CODEN: ZOKHA4; ISSN: 0044-460X

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB (CH2Cl)2 extraction of the upper parts of the plant in the presence of 10% NH4OH, treatment with 10% H2SO4, addition of NH4OH, and extraction with CHCl3 gave

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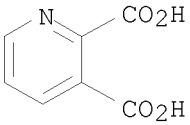
the crude alkaloid, which, rubbed with Me2CO yielded (from 5 kg. plant matter) some 20 g. makrotomine, C15H27O5N, m. 95-7°, [α]D -6.9°, picrate, m. 130-2° (from EtOH). Sapon. with 2N NaOH gave Me2CO and trachelanthamidine (cf. C.A. 41, 3092b), as well as tarry materials. Sapon. with Ba(OH)2 gave the same result. Makrotomine (3 g.) with KIO4-H2SO4 at room temperature gave AcH, (CO2H)2, trachelanthamidine, and Me2CO. Trachelanthamidine is not affected by HIO4. Thus makrotomine is an ester of trachelanthamidine with some acid which decompose on hydrolysis and forms Me2CO. Since HIO4 oxidation requires 1 mole O per mole alkaloid and the products are those cited above, the alkaloid is the ester of 2,3-dihydroxy-2-(1-hydroxyethyl)-3-methylbutyric acid with hexahydro-3-h-pyrrolo [1,2-al pyrrole-1-methanol.

IT 89-00-9P, Quinolinic acid
RL: PREP (Preparation)
(formation of, by oxidation of 5,8,9,10,11,12-hexahydro-6H-pyrido[3,2-a]quinolizine)

RN 89-00-9 HCAPLUS

CN 2,3-Pyridinedicarboxylic acid (CA INDEX NAME)



L27 ANSWER 14 OF 14 HCAPLUS COPYRIGHT 2009 ACS on STN
ACCESSION NUMBER: 1953:778 HCAPLUS
DOCUMENT NUMBER: 47:778
ORIGINAL REFERENCE NO.: 47:134d-i,135a-e
TITLE: Pyridine syntheses. I. Some reactions of "ene amines" with 1, 3-dicarbonyl derivatives
AUTHOR(S): Bottorff, Edmond M.; Jones, Reuben G.; Kornfeld, Edmund C.; Mann, Marjorie J.
CORPORATE SOURCE: Lilly Research Labs., Indianapolis, IN
SOURCE: Journal of the American Chemical Society (1951), 73, 4380-3
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 47:778

AB cf. C.A. 46, 983b. Condensations of ene amines, MeC(NH2):CHCN (I), MeC(NH2):CHCO2Et (II), MeC:NHCH2COMe (III), with EtOCH:C(CO2Et)COCO2Et (IV), EtCOC(:CHOEt)COCO2Et (V), and related compds. were carried out in an attempt to prepare 2,3,4,5-tetrasubstituted pyridines suitable for conversion to vitamin B6. Instead of the desired compds., the pyridines obtained were invariably substituted in the 2, 3, 5, 6-positions. IV (89 g.) and 62 g. II heated several hrs. on the steam bath and the product distilled in vacuo yielded 26 g. di-Et 2, 6-dimethyl-3, 5-pyridinedicarboxylate (VI); the residue on extraction with hot petr. ether (60-8°) left 2.5 g. white crystals, probably 3-acetyl-5-carbethoxy-6-methyl-2-pyridone (VII), m. 210-13°; the cooled petr. ether filtrate gave a solid (10.5 g.), m. 102.5-3.5°,

probably EtO2CC(Ac): CHNHC(:CMe2)CO2Et (VIII). The same experiment with 13 g. II and 16 g. HOCH:C(CO2Et)COCO2Et (IX) let stand 12 days at room temperature yielded 7.5 g. VI and 6.7 g. VIII. VI (35 g.) and 18 g. KOH refluxed 45 min. in 500 cc. absolute EtOH, filtered, the filtrate evaporated, the dried residue (24 g.) and 47 g. CaO in 40 cc. water distilled with a free flame, the distillate extracted with Et2O, the Et2O evaporated, and the residue distilled yielded 2, 6-lutidine, b. 139-41°; picrate, m. 100.5-102°. The di-K salt of the free acid of VI and 30 g. KMnO4 heated 4 hrs. on the steam bath in 500 cc. water, the mixture filtered, the filtrate evaporated to dryness in vacuo, and the residue let stand 24 hrs. in 300 cc. MeOH saturated with HCl yielded tetra-Me 2, 3, 5, 6-pyridinetetracarboxylate (X), m. 118-19° (from Et2O-Me2CO). IV (49 g.) in 50 cc. dry Et2O treated with 20 g. I, the mixture heated 30 min. on the steam bath, the liquid in 100 cc. Et2O washed with dilute Na2CO3 and water and dried, the Et2O evaporated, and the residue distilled yielded 36 g. di-Et 5-cyano-6-methyl-2,3-pyridinedicarboxylate (XI), b0.8 150°, b1 155°, nD25 1.5123, d2525 1.1708. Similar expts. in AcOH and absolute EtOH yielded 72 and 70%, resp., XI, b0.4 145-6°. XI (6.2 g.) and 4 g. NaOH refluxed 3 hrs. in 25 cc. water and 10 cc. EtOH and the solution digested with 200 cc. EtOH yielded 6.0 g. Na salt (XII) of 6-methyl-2, 3, 5-pyridinetricarboxylic acid. XII (5.8 g.) in 150 cc. water treated with 6.32 g. KMnO4 in 100 cc. hot water, the solution heated overnight on the steam bath, filtered, evaporated to dryness, and esterified with MeOH and HCl yielded 2 g. X, m. 118-19°. XII (3 g.) in 100 cc. MeOH saturated with HCl let stand 24 hrs. yielded the tri-Me ester (XIII), m. 78.5-9.5° (from Et2O). XII yielded the tris(p-bromophenacyl) ester, m. 190-2° (from dioxane-EtOH-water). IV (32 g.) in 25 cc. Et2O treated with 18 g. II, the mixture heated 30 min. on the steam bath, and distilled yielded 36 g. tri-Et 6-methyl-2, 3, 5-pyridinetricarboxylate (XIV), b0.5 160°, nD25 1.500, d2525 1.168. XIV sapond. with NaOH and esterified with MeOH and HCl yielded XIII, m. 78.5-9.5°. III and IV yielded 65-70% di-Et 5-acetyl-6-methyl-2,3-pyridinedicarboxylate (XV), b0.5 165-7°, m. 62-3°. XV (1 g.) moistened with alc., treated with 3 cc. 12 N NaOH, the mixture warmed a short time, diluted with 10 cc. water, and acidified with HCl yielded the free acid (XVI), m. 165-6° (decomposition) (from water). XVI (1 g.) treated with 25 cc. cold 5 N NaOH containing 1 g. Cl, the mixture let stand 1 hr., warmed 1 hr. on the steam bath, evaporated to dryness in vacuo, and the residue treated with MeOH containing HCl yielded XIII. IV (24.5 g.) in 100 cc. Et2O treated with 18 g. MeC(NH2):CHCONHPh, the solution let stand overnight, diluted with 200 cc. petr. ether, and chilled yielded 23 g. di-Et 5-carboxanilido-6-methyl-2,3-pyridinedicarboxylate, m. 121-2° (from C6H6-petr. ether). V (21.3 g.) and 10 g. I in 50 cc. Et2O yielded 11 g. Et 3-acetyl-5-cyano-6-methyl-2-pyridinecarboxylate (XVII), b0.8 132-7°, m. 94.5-95° (from Et2O-petr. ether). XVII shaken with 5 N NaOH and the solution acidified with HCl yielded the free acid (XVIII), m. 154-6° (decomposition) (from water). XVIII treated with NaOCl, hydrolyzed, and esterified yielded XIII. V (21.4 g.) and 13 g. II yielded 23 g. di-Et 3-acetyl-6-methyl-2,5-pyridinedicarboxylate (XIX), b2.5 180-5° m. 67-8°. XIX on sapon. yielded the free acid (XX), m. 210-13° (decomposition) (from water). XX on treatment with NaOCl yielded XIII. V (125 g.) and 58.5 g. III in 275 cc. Et2O let stand overnight, the solid filtered off, heated to boiling in 100 cc. EtOAc, and chilled yielded 37.5g. Et

β-[(1-methyl-3-oxo-1-butenylamino)methylene]-α, γ-dioxo-valerate (presumably), m. 164-5° (from EtOAc and C6H6petr. ether). The combined filtrates evaporated to dryness in vacuo, the residue in warm Et2O diluted with petr. ether until cloudy and chilled yielded 38.5 g. Et 3, 5-diacetyl-6-methyl-2-pyridinecarboxylate, m. 96-7° (from Et2O-petr. ether); free acid (XXI) m. 139-40° (decomposition) (from water). XXI on oxidation yielded XIII. CF3CO(:CHOEt)CO2Et (18 g.) and 11 g. II yielded 19 g. di-Et 2-methyl-6-trifluoromethyl-3, 5-pyridinedicarboxylate, b1 115-17°, nD25 1.4647, d2525 1.261.

IT 113052-06-5P, Quinolinic acid, 5-acetyl-6-methyl-
RL: PREP (Preparation)
(preparation of)

RN 113052-06-5 HCAPLUS

CN 2,3-Pyridinedicarboxylic acid, 5-acetyl-6-methyl- (CA INDEX NAME)